Fabrication and Characterization of Super Hard TiCrN Coating for Mechanical Application of Micro Drill Bits using Pulsed Magnetron Sputtering

Jayaprakash R, Ganeshmoorthy V, Vadivel S, Department of MechanicalEngineering, SethuInstituteofTechnology, Pulloor, Kariapatti, India

ABSTRACT

Nano structured TiCrN films were coated on Micro drill bits by pulsed reactive DC magnetron sputtering technique. We have also deposited nano structured TiCrN coatings on silicon wafer and on stainless steel substrates. The process parameters have been optimized to achieve highly adherent good quality TiCrN coating. The crystal structure, chemical composition and morphology of the TiCrN films were characterized by X ray diffraction, Atomic force Microscope, Scanning Electron Microscope and energy Dispersive analysis of X ray (EDAX). X ray diffraction patterns and Structural morphology of SEM results of different frequencies of power supply was compared.50 holes are drilled on 1mm thick 316L stainless steel plates by means of both the coated and uncoated drill bits. The holes are compared for its diametral precision, Chip removal and delamination area.

Keywords: General Characterization, Micro Drill Bits, Nano structured TiCrN coating, Pulsed Magnetron Sputtering.

1.0 Introduction

In recent years, there have been considerable advances in the development of hard and wearresistant coatings for applications in the field of cutting tools and other moving parts to improve durability (Stappen et al 1995). Transition metal nitrides such as titanium nitride (TiN), chromium nitride (CrN) and titanium– aluminum nitride (TiAlN) have been widely used as protective super hard coatings to increase the lifetime and performance of the cutting and forming tools (Horling et al 2005; Reiter et al 2006; Endrino et al 2006). These coatings have also shown their ability to improve the corrosion resistance of a variety of steel substrates (William Grips et al 2006). Addition of Al into TiN (or CrN) has been shown to improve the oxidation resistance of transition metal nitrides considerably (Barshilia and Rajam 2004a; Barshilia et al 2005, 2006). The structure and properties of TiN have been studied in detail by several researchers.Recently, the ternary nitride films such as TiCrN [6-11], CrZrN [12], TiAlN

[13], and CrAlN [14] are greatly interesting to enhance the mechanical and chemical properties at high temperature. TiCrN, especially, has been attracting more attention owing to high hardness, high temperature oxidation resistance, and low friction coefficient [6-11, 15-21]. Actually, it is well known that the film properties strongly depend on the microstructure of the films such as phase, grain, and defect structure which relate to the deposition parameters. In general, TiCrN films can be prepared by different techniques such as reactive magnetron sputtering, ion beam assisted deposition, cathodic arc, and electron beam evaporation. Among these methods, the reactive magnetron sputtering is one of the most widely used techniques to prepare the films with large area uniformity and strong adhesion. Although there are many research works regarding the structural and mechanical properties of TiCrN films, few studies reported the influence of the deposition parameters on chemical state and electronic structure of the TiCrN films. In this paper, to achieve dense and defect free TiCrN coatings, nano structured TiCrN coatings on Si Wafers using an indigenously developed cost effective pulsed reactive direct current magnetron sputtering process.

2.0 Experimental Procedure

The sputtering system consists of magnetron cathodes, the substrate holder, the feed-through, etc. High vacuum pumping system with a turbo-molecular pump was used to create a vacuum in the chamber of the order of 2.0 x10-5 mbar. Two direct-cooled unbalanced magnetron cathodes with feed-through mounted vertically in opposed-cathode configuration are used for cosputtering. The guns which holds the Ti target was powered by unipolar-pulsed DC generator and the Cr target was powered by D.C generator. The substrates were mounted on a substrate holder plate with planetary rotation and heating facility. This ensures uniform deposition of complex shaped substrates. A DC power supply was used for substrate bias and ion bombardment. Si (100) wafers (10mm \times 10mm) were cleaned ultrasonically in distilled water and acetone. In order to deposit TiCrN coatings Ti (purity 99.95%) target and Cr (purity 99.99%) target were sputtered in high purity Ar (99.999%) and N 2 (99.999%) plasma. The diameter of the sputtering targets was 50mm. The sputtering was done at a total Ar &N2 gas pressure of 5x10 - 3 mbar. The flow rates of Ar and N2 gases were controlled separately by mass flow controllers. The substrate to target distance was approximately 7 cm. The coatings were deposited at a substrate temperature of approximately 400 °C. The pulsed generators was operated at frequencies of 0 and 10 kHz and with a power of 160w and the D.C generators was operated with a power of 60w.. The thickness of the coating was 2µm. The structural analysis of the TiCrN films were done by X- ray diffraction (XRD: PANalytical, PW3040/60 X'pert PRO) using a monochromatic Cu K radiation and generator settings of 40 kV and 40mA. The XRD patterns were acquired in a continuous mode, scanning speed of $2^{\circ}/\text{min}$.

Table 1. Deposition conditions for TiCrN films.

Parameters

Details

Base Pressure (mbar)	$2x10^{-3}$
Working Pressure(mbar)	5×10^{-3}
Ti Sputtering Power, P _{Ti} (w)	160
Cr Sputtering Power, PCr (w)	60
Sputtering Temperature (°C)	400
Frequency (kHz)	0,1
Duration (min)	120

The elemental composition of the coatings was determined using energy dispersive X-ray analysis (EDAX) attached to SEM. The surface and structural morphologies were examined by atomic force microscopy and Scanning Electron Microscopy (Hitachi, S-3000H).

3.0 Results and Discussions

The X-ray diffraction patterns of TiCrN films deposited at various frequencies was shown in the fig.1. After deposition, the ferrite peaks are no longer detected and corresponding TiN, CrN, or TiCrN diffraction peaks can be observed. For TiN film, the (111), (200), (220) and (222) planes exhibited diffraction angles (2 θ) of about 36.6°, 43.6°, 63.5°,74.5° and 79.6°, respectively. Since CrN had the same crystalline structure as TiN but with a different lattice constant, those diffraction peaks were 37.0°, 43.6°,63.3° and 79.6° respectively.

The TiCrN film exhibited a similar XRD pattern as those of TiN or CrN. Initially coating at a frequency 0kHz two peaks are formed at an angle of 36.6° and 79° .Further increase in the frequency of the pulsed D.C. generators to 10kHz additional peaks arises at an angle of 43.6° and 63.5° . All the peaks with their corresponding hkl values are noted. The structural morphology of the TiCrN films with different conditions of Ti sputtering frequencies was examined by SEM as shown in Figure 2. The results revealed that these films showed globular structure and as the frequency increased 10 kHz, the formation TiCrN particles becomes much denser. The elemental composition of the TiCrN films was investigated by EDAX was show in the fig.2. As seen in the table 2, it shows the content of materials available on the coating in % wt. Overall Ti content in the coating was 23.02 % and chromium is 3.55 %. The sample used for this testing is TICrN coated on Si wafer.

Materials	Elemental Composition at %
Titanium	23.02
Chromium	3.55
Silicon	38.78
Nitrogen	34.65

As the coating is very thin, the Si wafer was also exposed which gives a Si weight % of 38.78 and the presence of N on the coating is 34.65%.

The presence of Titanium and chromium in the films is approximately the ratio of 6.5:1. The AFM surface images of the TiCrN films as a function of Ti sputtering frequencies are shown in Figure 3. It was found that the films have rough surfaces and sphere like TiCrN particles were formed as same as the SEM images.

However, it is seen that the surface morphologies of the films deposited at various sputtering frequencies are significantly different. As the increased from 0 to 10 kHz, the root-mean- square roughness (rms) increased from 1.33 to 1.51nm.

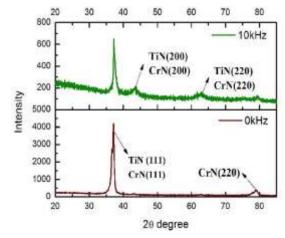
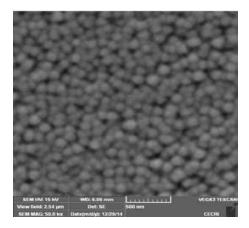


Fig. 1 XRD Pattern of TiCrN coating



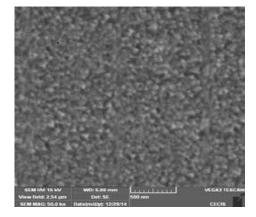


Fig. 2. Structural SEM morphology of TiCrN films deposited on different frequencies (a) 0, (b) 10 kHz

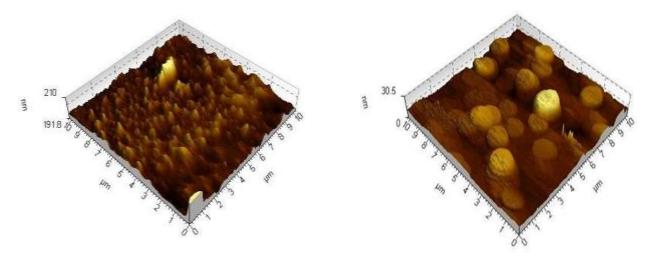


Fig. 3. AFM data of TiCrN coating deposited on Si wafers at sputtering frequency of (a) 0KHz, (b) 10KHz

4.0. Comparative Studies on Micro Drill Bits

Nano structured TiCrN was coated with optimized coating parameters to achieve good quality adherent coating was made on micro drill bits.Fig.7 shows the image of TiCrN coated micro drill bit.



Fig. 4. TiCrN coated micro drill bit

The coated and uncoated drill bits are tested on 1mm thick 316L stainless plates. 50 holes are drilled on plates by both the drill bits. The images of 1st and 50th holes are recorded by means of Rapid-I machine vision system. Diameter precision of the hole, delamination and chip removal of the both drills are compared.

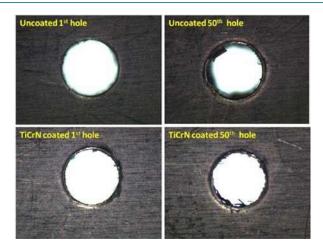


Fig.5 Holes drilled by TiCrN and uncoated micro Drill bit.

When drilling stainless steel, the built up edge is either absent or is very small with TiCrN coated drill bit. The surface finish is greatly improved by elimination of built up edge with the TiCrN coated drill bit. The quality of the drilled hole was significantly better than uncoated drill bit. The surface finish, diameter precision of the hole, reduced material build up on the cutting edges and finally increased tool life were obtained with TiCrN coated HSS drill bits. The material build up on the margin of the drill bit leads to poor surface finish of the drilled hole. It was possible to effectively overcome the formation of material build up on the area of the cutting edge and smoother flow of the chip over the coated flutes. With uncoated drill bit, it was not possible to effectively overcome the formation of material build up on the area of the cutting edge.

5.0 Conclusion

Nano structured TiCrN films were deposited by Pulsed DC Magnetron sputtering technique. The effects of the Ti sputtering frequency on the chemical state, electronic structure, crystal structure, and morphology of the films were investigated. The XRD results shows that with increase in frequencies from 0 to 10 kHz additional peaks are seen which means that the addition deposition of excess TiCrN particles on to the substrate. The SEM result shows that globular structures of TiCrN particles are formed. Elemental Composition of Titanium to Chromium on the film is approximately in the ratio of 6.5:1. Increased presence of Ti is because of pulsed D.C power supply given to Ti Target. AFM images showed the roughness of the film surfaces. An improved diameter precision and chip removal was found by drilling with TiCrN coated drill bits and the delamination area was also considerably reduced.

References:

- a) Barshilia H C and Rajam K S, Bulletin of Materials Science (2004a), vol 2735, pp 607-614.
- b) Horling A, Hultman L, Oden M, Sjolen J and Karlsson L, Surface Coating Technology (2005), vol. 191, pp 384-392.
- c) Stappen M V, Stals L M, Kerkhofs M and Quaeyhaegens C Surface Coating Technology (1995), vol. 629, pp74–75.
- d) William Grips V K, Barshilia H C, Ezhil Selvi V and Rajam K S, Thin Solid Films (2006), vol. 514, pp204-210.
- e) P. Hones, R. Sanjin'es, and F. L'evy, Thin Solid Films(1998), vol. 332, no. 1-2, pp. 240–246.
- *f) H. S. Choi, D. H. Han, W. H. Hong, and J. J. Lee, Journal of Power Sources, (2009),vol.* 189 ,no. 2, pp. 966–971.
- g) D. H. Jung, H. S. Park, H. D. Na, J.W. Lim, J. J. Lee, and J.H.Joo, Surface and Coatings Technology(2003), vol. 169-170, pp.424–427.
- h) J. G. Han, H. S. Myung, H. M. Lee, and L. R. Shaginyan, Surface & Coatings Technology (2003), vol. 174-175, pp. 738–743.
- i) J.H.Hsieh, W.H. Zhang, C. Li, and C. Q. Sun, Surface and Coatings Technology (2001), vol. 146-147, pp. 331–337.
- *j)* P. Hones, R. Sanjin'es and F. L'evy, Thin Solid Films (1998), vol. 332, no. 1-2, pp. 240–246.
- *k) Y. Otani and S. Hofmann, Thin Solid Films*(1996), *vol.* 287, *no.* 1-2, *pp.* 188–192.
- *l)* Z. G. Zhang, O. Rapaud, N. Bonasso, D. Mercs, C. Dong and C. Coddet, Vacuum(2008), vol. 82, no. 11, pp. 1332–1336.
- m) K. Chu, P. W. Shum, and Y. G. Shen, Materials Science and Engineering B (2006), vol. 131, no. 1–3, pp. 62–71.
- n) L. Wang, G. Zhang, R. J. K. Wood, S. C. Wang, and Q. Xue, Surface and Coatings Technology(2010), vol. 204, no. 21-22, pp. 3517–3524.
- o) D. B. Lee, Y. C. Lee, and S. C. Kwon, Surface and Coatings Technology(2001), vol. 141, no. 2-3, pp. 232–239.
- *p)* S. M. Aouadi, K. C.Wong, K. A. R. Mitchell et al., Applied Surface Science (2004), vol. 229, no. 1–4, pp. 387–394.
- *q)* J. J. Nainaparampil, J. S. Zabinski, and A. Korenyi-Both, Thin Solid Films (1998), vol. 333, no. 1-2, pp. 88–94.
- r) J. Vetter, H. J. Scholl, and O. Knotek, Surface and Coatings Technology(1995), vol. 74-75, no. 1, pp. 286–291.
- s) K. H. Lee, C. H. Park, Y. S. Yoon, and J. J. Lee, Thin Solid Films (2001), vol. 385, no. 1-2, pp. 167–173.
- t) K.H. Lee, C.H. Park, Y. S. Yoon, H.A. Jehn, and J. J. Lee, Surface and Coatings Technology (2001), vol.142–144, pp. 971–977.
- u) V. M. Vishnyakov, V. I. Bachurin, K. F. Minnebaev et al., Thin Solid Films (2006), vol. 497, no. 1-2, pp. 189–195.